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ABSTRACT

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Transition Regions	2					
Controlled Atmosphere	5					
Poly(norbornadiene)	1					

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II

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Technical Report No. 12

THERMOMECHANICAL BEHAVIOR OF A POLY(NORBORNADIENE)

by

J.K. Gillham, M. B. Roller and J. P. Kennedy

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Polymer Materials Program

Chemical Engineering Department

Princeton, New Jersey 08540

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III

THERMOMECHANICAL BEHAVIOR OF A POLY(NORBORNADIENE)

M. B. Roller and J. K. Gillham
Polymer Materials Program
Department of Chemical Engineering
Princeton University
Princeton, New Jersey 08540

J. P. Kennedy
Institute of Polymer Science
The University of Akron
Akron, Ohio 44325

ABSTRACT

A high temperature polyolefin, synthesized cationically from norbornadiene [bicycls-(2,2,1)-heptadiene-2,5], was studied thermomechanically with respect to physical transitions and stability in nitrogen. The glass transition was found to be 320°C, the highest known for a purely hydrocarbon addition polymer. The thermomechanical technique of Torsional Braid Analysis, Thermogravimetric Analysis, Differential Thermal Analysis, Infrared studies, and solubility studies were used to investigate the sequential events of the glass transition and degradation. The polymer is of particular interest in being a high temperature plastic which should be processible at high speeds in an inert atmosphere, but the presence of tertiary hydrogen atoms would confer auto-oxidative properties.

IV

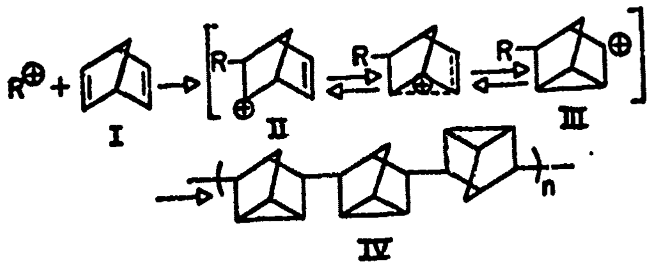
THERMOMECHANICAL BEHAVIOR OF A POLY(NORBORNADIENE)

M. B. Roller and J. K. Gillham
Polymer Materials Program
Department of Chemical Engineering
Princeton University
Princeton, New Jersey 08540

and J. P. Kennedy
Institue of Polymer Science
The University of Akron
Akron, Ohio 44325

INTRODUCTION

The cationic polymerization of norbornadiene [bicyclo-(2,2,1)-heptadiene-2,5 (I below)] has been reported to lead to predominantly structure IV (1,2). UV, IR and NMR studies corroborated this expectation (1,2). The cationic reaction mechanism which was proposed involves a transannular rearrangement (II → III) of the electrons of the initially formed addition intermediate (II), prior to addtion of another norbornadiene monomer (I) (1,2).



2,7 enchainment was excluded for steric reasons. The mechanism was not stereospecific and therefore the product was atactic and amorphous [which was confirmed by X-ray examination (1,2)]. However, an examination of models would lead one to expect bonds joining repeat units to be equatorial to equatorial. The three dimentional cage structure in the polymer would confer a high degree of chain stiffness and therefore a high glass transition. The presence of a highly strained and substituted cyclopropane ring in the cage structure cast some doubt on how thermally stable the polymer might be, while the presence of tertiary hydrogens would be expected to lead to oxidative instabilities.

The present report presents a preliminary examination of this polymer's thermomechanical behavior and thermal

stability. The techniques used were: Torsional Braid Analysis (TBA) for the thermomechanical spectra (3,4,5); Differential Thermal Analysis (DTA), to monitor heat effects; Thermogravimetric Analysis (TGA), to study weight loss; Infrared Analysis (IR) to monitor chemical changes; and solubility studies to further examine thermal effects via crosslinking. All the studies were carried out with a heating rate of 2 deg.C/Min with the exception of DTA ($\Delta T/\Delta t$ = 20 deg.C/Min), and all were performed in a dried nitrogen atmosphere (TGA was also studied in air).

SYNTHESIS

Norbornadiene [bicyclo(2,2,1)-hepta-2,5-diene] monomer (Matheson, Colman and Bell) was distilled before use. Gas chromatographic analysis indicated greater than 98 percent purity and the presence of four or five minor impurities.

Polymerizations were carried out in a dry box in stirred glass reactors using a published procedure (1). The catalyst, aluminium chloride, was dissolved in ethyl chloride (2.5 percent) and was added slowly (1-2ml every 2-3 min.) to the reactor. The reactor contained a homogeneous solution of norbornadiene (19.8g) in ethyl chloride (130ml) at -127 deg.C. The temperature of the reaction medium increased to no higher than -125.5 deg.C during the course of the synthesis (>1 hour). Polymerization started immediately after catalyst introduction. The reaction was terminated by introducing precooled n-propanol. The product was washed thoroughly with methanol, filtered and dried under nitrogen at 50deg.C. The yield was 2.50g or 12.5 percent. The material was soluble in toluene, benzene, ether and carbon tetrachloride. Its number average molecular weight ($\bar{M}n$ via vapor phase osmometry) was 9850, indicating a degree of polymerization of about 100. $\bar{M}n$=14970 and $\bar{M}w/\bar{M}n$=3.73 by gel permeation chromatography, when treated as polystyrene.

EXPERIMENTAL

The thermomechanical data were determined throughout the range -180 to +500 deg.C by the technique of torsional braid analysis which uses a modified torsional pendulum operating at about 1 cps. The Torsional Braid Analyzer is a free hanging torsional pendulum with the specimen consisting of a multifilamented (3600) glass braid impregnated with the sample polymer. The specimen is fabricated *in situ* by removing solvent from a braid that has been soaked in a polymer solution before mounting. The specimen for the pendulum was made using a 10 percent (wt./vol.) solution of the polymer in benzene and a glass braid support. Solvent was removed from the composite specimen by heating (2 deg.C/Min) to 200 deg.C and cooling. Attached to the

lower clamp is a polarizer disc, the inertial mass, which when coupled with another polarizer over a photocell, acts also as a "linear-with-angle" transducer to convert the mechanical oscillations into electrical signals (5). The mechanical oscillations and the analogue signals approximate damped sine waves. The mechanical parameters are deduced from the character of the analogue signals. For isotropic, homogeneous specimens subject to small stains, $G' = K (1/P^2)$, where G' is the in-phase elastic modulus, P = the period of oscillation and K is a constant dependent upon geometry; the logarithmic decrement, a measure of the ratio of energy dissipated to maximum energy stored on mechanical deformation is defined as: $\Delta = \log(A_1/A_2) = \log(A_3/A_4)\ldots = \log(A_n/A_{n+1})$. Due to composite nature, the small size and the irregular geometry of the specimens, the work discussed herein is presented in terms of the Relative Rigidity, = $(1/P^2)$, replacing G'. The logarithmic decrement (Δ) is presented as the mechanical damping index, = $1/n$, where n is the number of oscillations between two fixed but arbitrary boundary amplitudes (e.g. A_i/A_{i+n} = 20; taken constant over any thermomechanical experiment); $1/n$ is directly proportional to the logarithmic decrement ($\Delta = 1/n \log[A_i/A_{i+n}]$).

The TGA'S were performed using a du Pont 950 Thermogravimetric Analyzer in nitrogen and air. The DTA was run on a Mettler Thermoanalyzer at 20 deg.C/Min in nitrogen

The TR study was performed on a single film enclosed between two NaCl crystals held together. The holder, salt plates and film were suspended in the TBA oven, in a nitrogen atmosphere, and were heated at 2 deg.C/Min to various "quench points" at which time they were removed from the oven to a nitrogen chamber held at room temperature, without exposure to air The quenched specimen was examined at room temperature by a grating infrared spectrophotometer, Perkin-Elmer Model 237B, at low scan and normal slit opening The specimen was returned to the nitrogen quench stream and purged before reintroduction to the oven at the quench point, after which the oven temperature was taken at 2 deg C/Min to the next quench point. The quench points were taken as significant temperatures in the thermomechanical spectrum.

The solubility studies were performed in a manner similar to the TR studies, except that separate film specimens were used, each taken at 2 deg.C/Min from room temperature to its quench point. The behavior of the thermally treated films in excess benzene was observed visually

THERMAL ANALYSES (TGA, DTA)

A sample of the solution used to fabricate the TBA specimen was examined on a du Pont 950 Thermogravimetric Analyzer with the same drying and run cycle as that used in the thermomechanical work. After drying at room temperature in nitrogen for 15 min. and then heating at 2 deg.C/Min to 200 deg.C, the specimen was cooled to room temperature and run at 2 deg.C/Min to 500 deg.C. The thermogram obtained was essentially the same as those obtained in runs made on the dry, powdered specimen (as recieved). The thermogram shown in Fig.1 shows a small, slow weight loss starting at 250 deg.C which reached 6 percent by 370 deg.C (in nitrogen). At about 410 deg.C the specimen started to lose weight rapidly; by 500 deg.C the rate of loss had diminished and the specimen had been reduced to about 29 percent of its original weight. The initial 6 percent weight loss need not have been degradative if the molecular weight distribution included appreciable amounts of low molecular weight species. Monomer, dimer and trimer, would have normal boiling points in the range of 100 deg.C to 350 deg.C (6). A low molecular weight tail is apparent in the GPC curve (Fig. 2). The large weight loss in the 420 to 460 deg.C range is due to polymer pyrolysis. In air, there was an initial weight gain beginning at 150 deg.C leading to a maximum weight of 102 percent of the original at 240 deg.C and a return to 100 percent by 275 deg.C. The presence of the weight maximum was due to the competitive effects of addition of oxygen (presumably to the formation of hydroperoxides at the tertiary hydrogen sites) and the removal of both low molecular weight oligomers and of low molecular decomposition products. Just above 280 deg.C there was an increase in the rate of oxidative weight loss preceding a region of moderating rate. At about 425 deg.C there was another increase in rate of loss which approximately corresponds to the thermal degradation noted in nitrogen. The specimen was completely oxidized by 500 deg.C and left no residue.

The DTA'S were performed in two stages. The dry powder was first taken at 2 deg.C/Min to 200 deg.C (to simulate the drying step in the TBA analysis) and then, after cooling, was taken at 20 deg.C/Min to 500 deg.C. In the vicinity of 300 deg.C an endothermic dip typical of the glass transition temperature was evident. The exact position of the glass transition temperature was difficult to determine. At 420 deg.C a large exotherm began. The data were somewhat ambiguous due to large baseline drift and therefore are not shown. The important point is that at 20deg.C/Min, the glass transition temperature and the degradation processes

were distinguishable.

THERMOMECHANICAL ANALYSIS

The thermomechanical behavior of the polymer in dried nitrogen was determined over the range -180 to 500 deg.C. The -180 to 350 deg.C curves in Figure 3 show the initial behavior of the specimen after it had been preheated to 200 deg.C (at 2 deg.C/Min) in order to remove the benzene solvent. The experiment was preceeded by cooling to -180 deg.C (at 2 deg.C/Min). The cooling and subsequent reheating data were not absolutely reproducible, although the damping peak positions and the curve shapes were similar. Only the reheating data are shown. In the low temperature region, there were multiple damping peaks with distinct maxima at -60 and -140 deg.C which were accompanied by changes in slope of the rigidity curve. At 225 deg.C there was a small shoulder in the broad glass transition damping peak that is also accompanied by a drop in modulus. The glass transition (Tg) is characterized by a large drop in rigidity and a large damping maximum at 320 deg.C. The DTA study previously cited indicated an endothermic shift in this region which is typical of Tg. The glass transition temperature region was found to be more pronounced(sharper loss peak and sharper region of rigidity decrease) after heating to 350 deg.C as shown by the cooling curves in Figure 3. The heating of the polymer to 350 deg.C simplified the pre-Tg region by eliminating the damping shoulder and drop in modulus in the 200-250 deg.C temperature range. Later studies, requiring the casting of films, showed that the polymer is a poor film former; heating above Tg probably improved the polymer coating on the braid. A new specimen was dried to 350 deg.C in nitrogen and then data was taken from 350 to -180 to 500 deg.C (Figure 4, curves 1). The data indicated that the low temperature transitions (unlike the previous specimen, the data were reversible on cooling from 350 deg.C and subsequent reheating) were not due to the formation of a poor film coating on the braid. On heating above 350 deg.C, the rigidity began to increase at about 370 deg.C , displayed a small maximum at 415 deg.C and then a large sigmoidal rise between 435 and 470 deg.C; after which it decreased slightly to 500 deg.C. The 435 deg.C increase in rigidity was accompanied by a damping peak at 453 deg.C after which the damping decreased sharply. The stiffening reactions were also detected as a large exotherm by DTA (described above). Visual examination of the cooled pyrolyzed specimen indicated that the polymer was converted into a smooth , glossy, black and opaque coating on the

braid Curve 2, Figure 4 represents the cooling behavior of another specimen after drying to 400 deg.C (2 deg.C/Min). Note that the glass transition shifted up 7 deg.C to 327 deg.C and the damping peak is narrower and less intense than for the previous specimen The increase in Tg and decrease in damping peak height are typical of the effect of increasing crosslink density in a given polymeric system (7) Narrowing of the damping peak is not typical , but is reasonable since some of the peak skewness may have been due to reactions occurring above Tg.

SOLUBILITY STUDY

A study was undertaken to determine the temperature region to which a film of the polymer must be heated ($\Delta T/\Delta t$ = 2 deg.C/Min) in order to become insolublized by chemical reactions (eg. crosslinking or chain stiffening). Seven solutions were prepared in small vials. The benzene in the solutions was removed at reduced pressure (23 in.Hg) overnight over the samples at 50 deg.C . One specimen was kept as a control The other dried solutions were suspended in the TBA oven and taken, one at a time, at 2 deg.C/Min from room temperature to various quench temperatures in nitrogen. The quench temperatures were room temperature (control), 100, 180, 260, 320, 360, and 400 deg.C. These temperatures correspond to significant points on either the TBA, DTA, or TGA plots. After quenching , excess benzene was added to the vial and after a day's standing they were compared to each other and to the control solution. The specimens were virtually unaffected until the 180 deg.C specimen though soluble, had begun to yellow. The yellow color of the solutions intensified without the formation of visible gel for specimens heated up to 360 deg.C. A small amount of swelled gel was evident in the 360 deg.C specimen, indicating that chemical crosslinking or chain stiffening occurred above Tg (as represented by the 320 deg.C specimen). A major portion of the 400 deg.C specimen was gelled and a brownish char was visible. The soluble portion of the 400 deg.C specimen displayed the same degree of yellowness as the 300 deg.C specimen. The exact concentrations of the solutions were not determined.

INFRARED STUDY

A qualitative infrared study was undertaken so as to correlate changes monitored by other techniques with chemical changes as revealed by the IR spectrum. A film of the polymer, formed by predrying from a benzene solution

slowly so as to prevent bubbling before drying at reduced pressure (23 in. Hg at 50 deg.C), was placed between two NaCl plates which were held together in an aluminum holder. An IR spectrum was run before any heat treatment (the IR spectrum in Figure 5 and the NMR spectrum in Figure 6 are presented for identification purposes and were not generated in this laboratory; they were provided by the ESSO Research and Engineering Company, Linden, N.J.). The scan corresponded well with the spectrum presented earlier (Reference 1 and Figure 5). (The band at 6.15 microns is water from KBr, and appears only in conjunction with the 2.8 micron band.) The specimen was placed in the TBA oven and heated at 2 deg.C/Min, in nitrogen, to several quench points: 100, 180, 260, 320, 370, 440, and 500 deg.C. Each IR scan was made after heating from the previous quench point to the new one. There were no apparent changes in the scans until 440 deg C when the 3050 cm^{-1} band, the 2875 cm^{-1} shoulder, the 1730 cm^{-1} band, the 1300 cm^{-1} band and the 800 cm^{-1} band decreased significantly relative to the 2925 cm^{-1} band. Several bands in the 1200 to 850 cm^{-1} region also seemed less intense. A new band appeared in the 1600 cm^{-1} region indicating possible unsaturation(8). The 800 cm^{-1} peak has been attributed to the 2,6-disubstituted nortricyclene structure(9,10,11). The changes observed for the 440 deg.C quench point would tend to indicate a major breakdown in the origional nortricyclene structure, but the large peak remaining in the 2925 cm^{-1} region suggests that a hydrocarbon structure still remains. The 500 deg.C sample scan displayed an almost total disappearance of all but the most prominant of the original peaks which were rendered weak.

CONCLUSIONS

The thermomechanical spectra of the olefinic polymer, poly(bicyclo-(2,2,1)-heptadiene-2,5) or poly(norbornadiene), indicate a glass transition temperature of 320 deg.C. This is the highest known Tg for a linear, soluble and fusible hydrocarbon polymer. It is also noteworthy that this polymer is formed without the evolution of volatile by-products (as with condensation polymers) and that utilizing suitable heating rates, the glass transition and subsequent degradation reactions can be separated. The onset of degradation at temperatures just above Tg is not unusual for high temperature plastics. At Tg the diffusive processes characteristic of the polymeric material increase by orders of magnitude and consequently the conditions are much more favo-able for initiating and sustaining chemical

reaction (5,12).

The presence of low temperature glassy state relaxations indicate that there is some mechanism of energy dissipation active at these low temperatures. Yet, the types of submolecular motions (crankshaft, rotations, oscillations) which are normally thought of as the origin of these relaxations are difficult to imagine with such a structure. This supports the notion that the more complex and stiffer a backbone structure, the broader and more complex the relaxation spectra (12).

It is also of interest that here is a high temperature polymer which sould be processible at high speeds, but since it contains many tertiary hydrogen atoms in its structure, should also be amenable to auto-oxidative degradation. In other words, poly(norbornadiene) is a potential high temperature plastic that due to its own structural features, should degrade entirely under long term exposure to the atmosphere.

ACKNOWLEDGMENT

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FIGURE CAPTIONS

Figure 1. Thermogravimetric analysis of poly(norbornadiene) in nitrogen and in air from 0 to 500 deg C at 2 deg. C/Min.

Figure 2 Gel permeation chromatogram of poly(norbornadiene).

Figure 3. Thermomechanical spectra of poly(norbornadiene) in nitrogen from -180 to 350 to 25 deg.C at 2 deg C/Min. The specimen was dried in nitrogen at 2 deg C/Min to 200 deg.C.

Figure 4. Thermomechanical spectra of poly(norbornadiene) in nitrogen (specimens dried to 350 deg.C at 2 deg.C/Min). Curve 1: 130 to -180 to 500 deg.C. Curve 2 is the cooling curve from 400 deg.C of a similarly preheated second specimen (data for curves 1 and 2, 130 to -180 to 400 deg.C were identical): 400 to -180 to 25 deg.C.

Figure 5. Infrared spectrum of poly(norbornadiene).

Figure 6. NMR spectrum (100MegHz) of poly(norbornadiene).

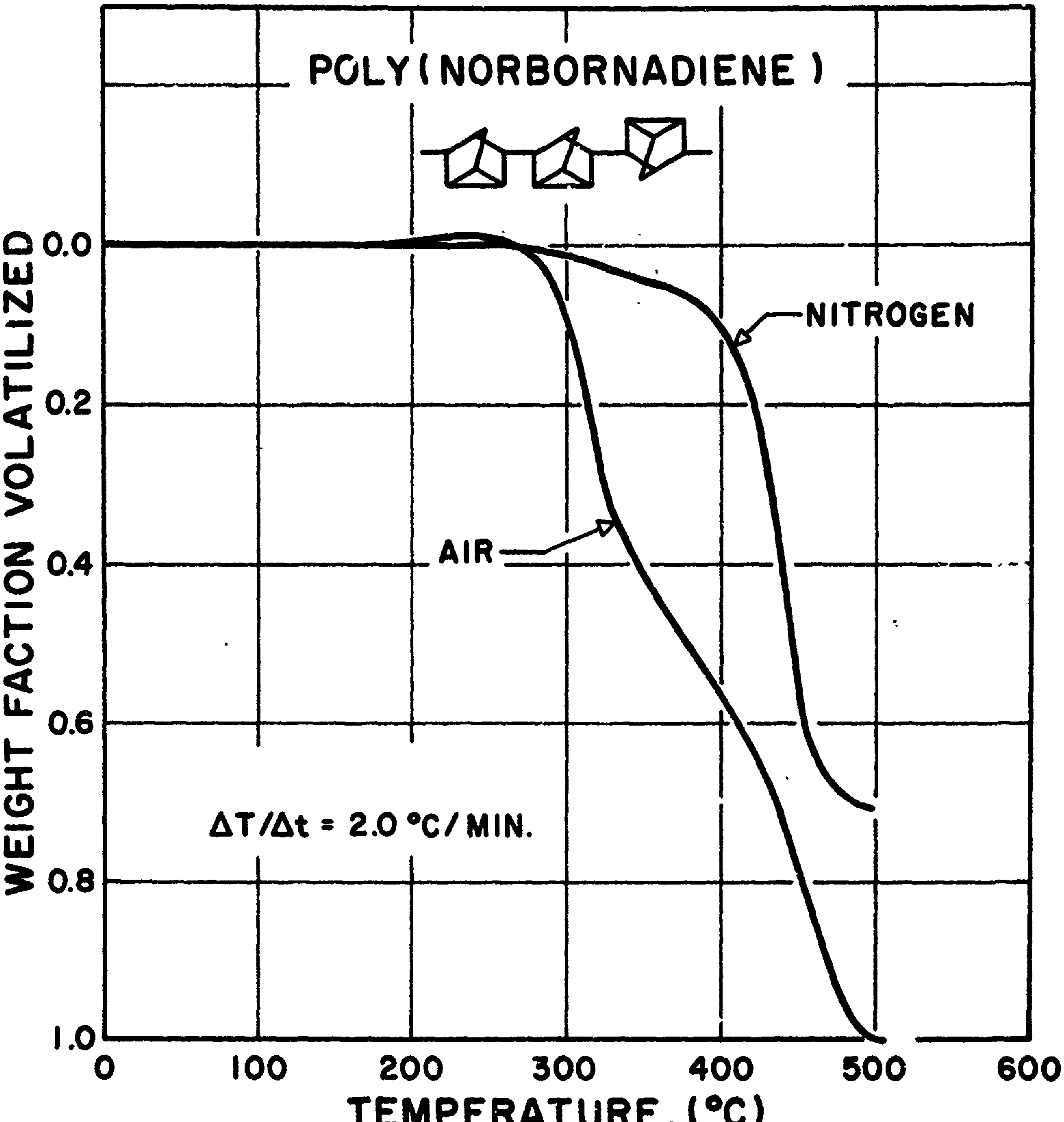

POLY(NORBORNADIENE)
WEIGHT FACTION VOLATILIZED
0.0
0.2
0.4
0.6
0.8
1.0
NITROGEN
AIR
ΔT/Δt = 2.0 °C/MIN.
0
100
200
300
400
500
600
TEMPERATURE, (°C)

FIGURE 1

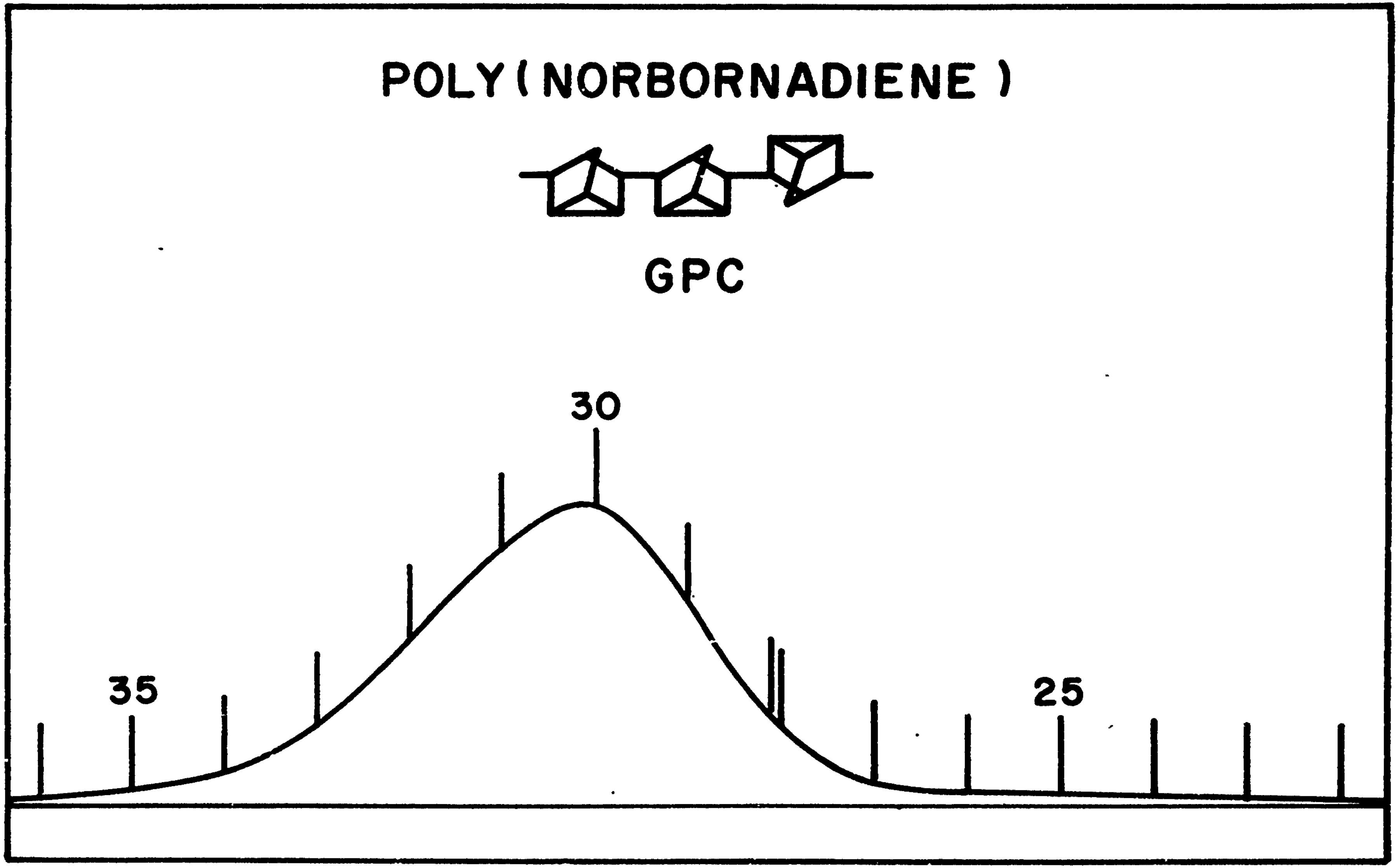


FIGURE 2

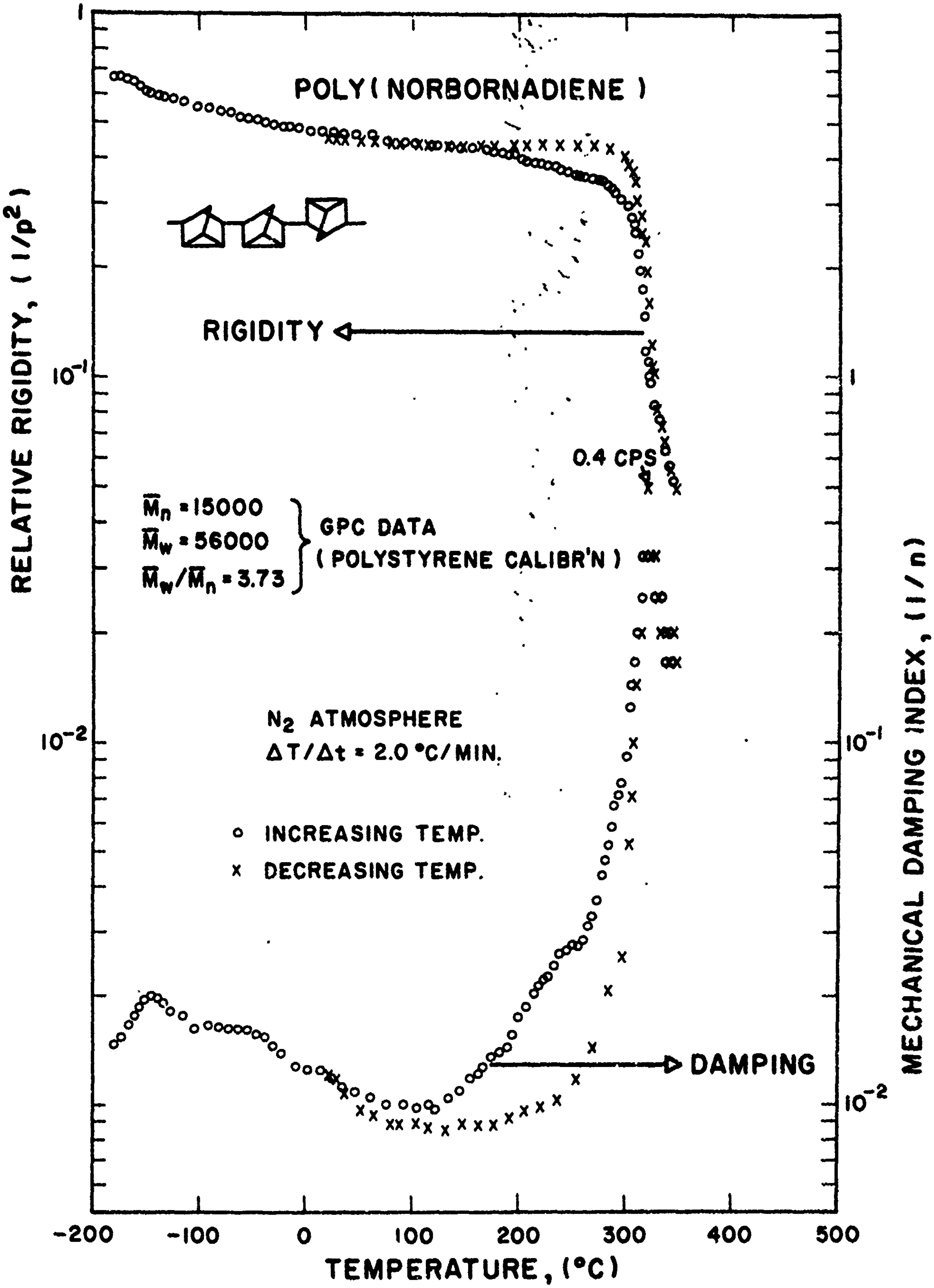
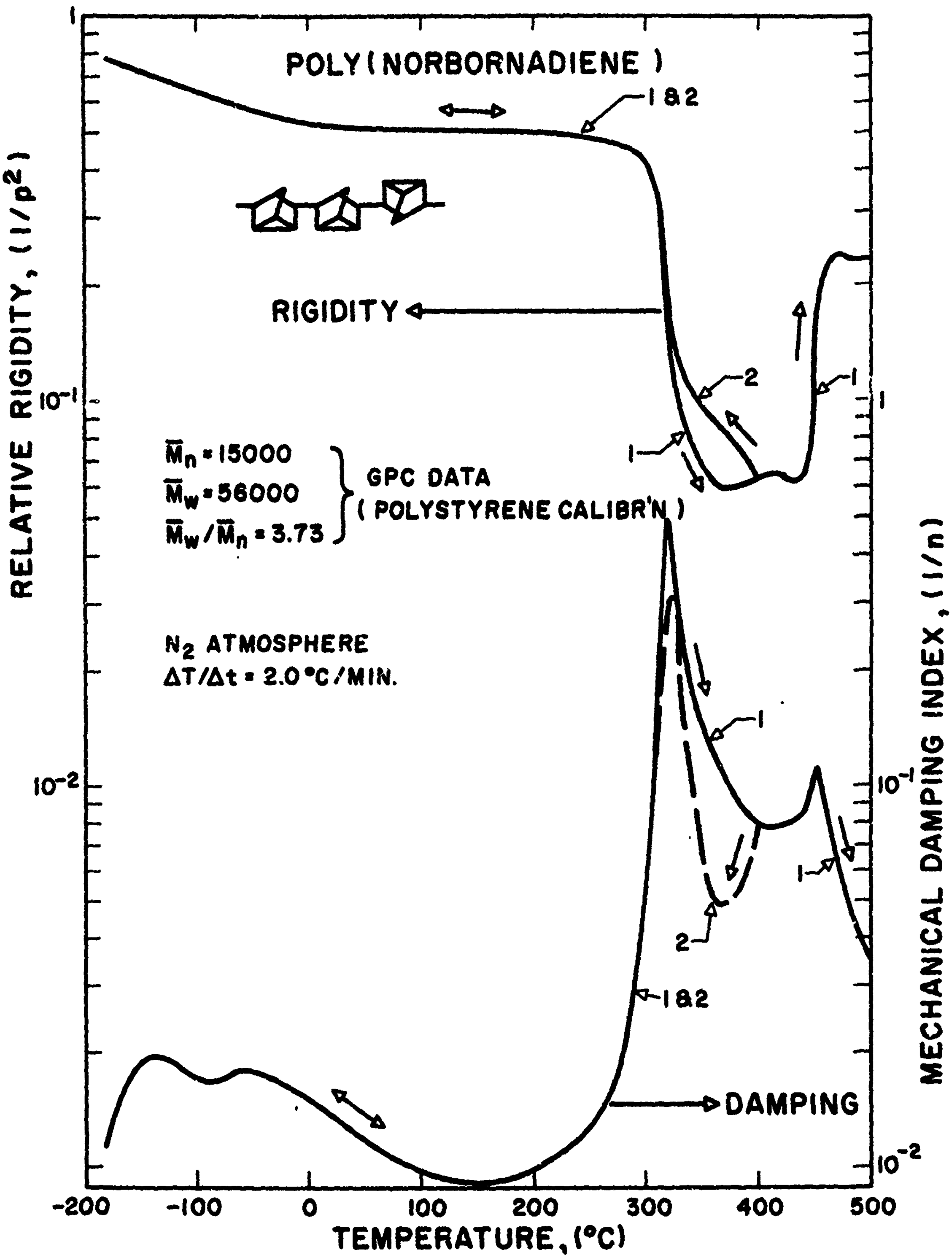
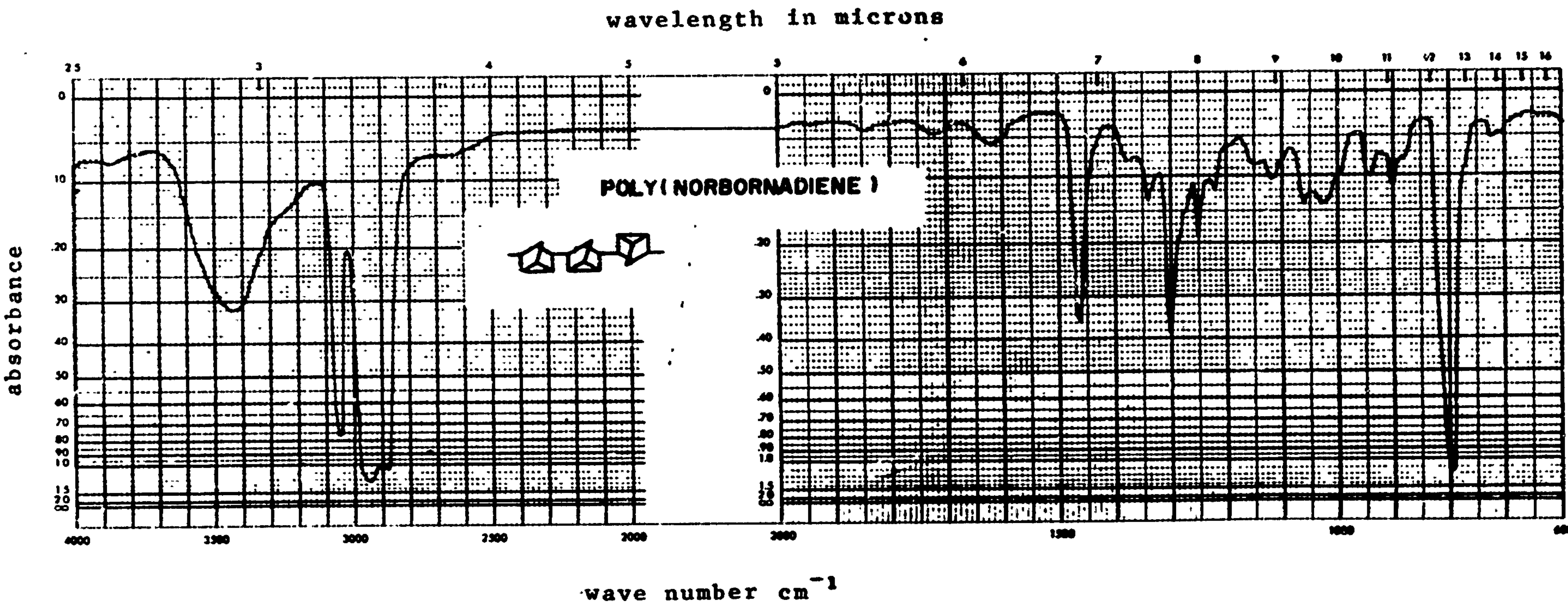

POLY (NORBORNADIENE)
RIGIDITY
0.4 CPS
$\overline{M}_n$ = 15000
$\overline{M}_w$ = 56000
$\overline{M}_w/\overline{M}_n$ = 3.73
GPC DATA
(POLYSTYRENE CALIBR'N)
N_2 ATMOSPHERE
$\Delta T/\Delta t$ = 2.0 °C/MIN.
o INCREASING TEMP.
x DECREASING TEMP.
DAMPING
RELATIVE RIGIDITY, ($1/p^2$)
MECHANICAL DAMPING INDEX, (1/n)
TEMPERATURE, (°C)
1
10^{-1}
10^{-2}
−200
−100
0
100
200
300
400
500

FIGURE 3



POLY(NORBORNADIENE)
1&2
RIGIDITY
2
1
1
$\overline{M}_n = 15000$
$\overline{M}_w = 56000$
$\overline{M}_w/\overline{M}_n = 3.73$
GPC DATA
(POLYSTYRENE CALIBR'N)
N_2 ATMOSPHERE
$\Delta T/\Delta t = 2.0\,°C/MIN.$
1
1
2
1&2
DAMPING
RELATIVE RIGIDITY, $(1/p^2)$
MECHANICAL DAMPING INDEX, $(1/n)$
1
10^{-1}
10^{-2}
1
10^{-1}
10^{-2}
-200
-100
0
100
200
300
400
500
TEMPERATURE, (°C)

FIGURE 4



wavelength in microns
POLY(NORBORNADIENE)
absorbance
wave number cm⁻¹

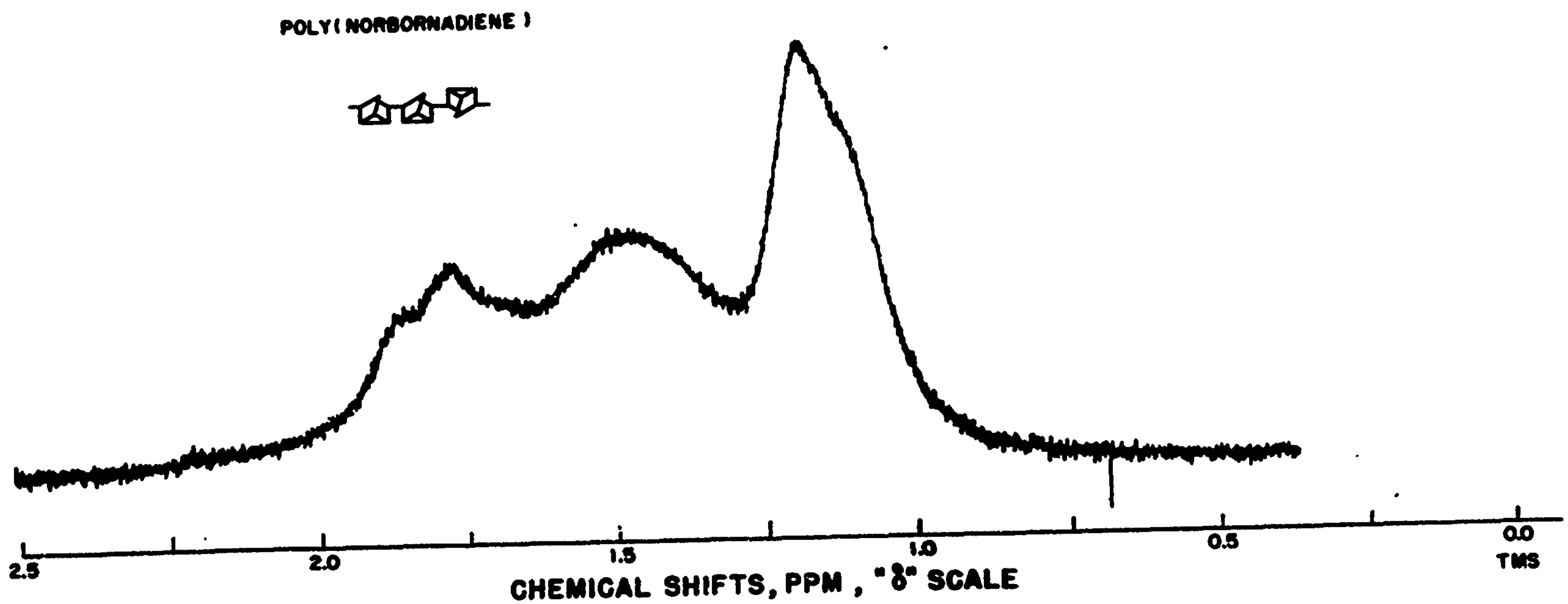

POLY(NORBORNADIENE)
2.5
2.0
1.5
1.0
0.5
0.0
TMS
CHEMICAL SHIFTS, PPM , "δ" SCALE

FIGURE 6